



Marked Up Version of Changes to the Specification

Adding solvent to the semi-solid viscous liquid molding resins useful as a binder produces binder solutions suitable for spraying. The [solvents] resins should have room temperature tack and be compatible with the infusion resin selected. For cyanate ester infusion resins, we typically use CIBA's M-20 semi-solid cyanate ester resin that is extremely tacky at room temperature. Some semi-solid resins with no room temperature tack can be used if they develop tack when heated, for example, 5250-4-RTM bismaleimide resin. The solutions sometimes require catalysts for resin activation. For more latent spray formulations, the catalysts can be eliminated or reduced from the mix to allow higher temperature vacuum debulk operations without adversely advancing the degree of cure of the binder. Binder contents can be increased at ply edges to provide greater dimensional integrity and less edge fraying. The binder might also incorporate thermoplastic or rubber toughening agents for improved damage tolerance and ballistic survivability.

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Although at least four times more expensive than similar aluminum tooling, PYREX glass project plates and tooling bars allow direct visual observation of the resin flow front as the preform is being infused. The leading edge of a resin wave front has a low angle tapered cross-section through the preform thickness. The infusion process goes through a cyclic fill and drain process if the flow rate in the vacuum tubes is not regulated to a low rate prior to final tube closure. Glass tooling is valuable for studying the infusion process and learning to control it, because the tooling allows visual inspection throughout the process. Glass [tolling] tooling, however, is likely impractical for many production processes.

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Almost all preform materials can be used without binders to produce simple flat panels or slightly contoured shells without ply tailoring. Complex shapes such as radomes, tailcones, integrally stiffened shells, deep structures, multi-directionally stiffened components or highly tailored structures that are built, require tackifiers for material adhesion to the tooling, enhanced consolidation or debulking, improved trimming, and better dimensional control.

A simplified and possibly improved M-20 binder solution appears technically feasible. Uncatalyzed M-20 cyanate ester can be thermally cured at temperatures above 250°F. Since the cyanate ester infusion resins are typically cured at 350°F and higher temperature postcures are often required, M-20 cure could be expected to cure in the absence of any catalyst. The catalyst used for the cyanate ester infusion resins can also probably catalyze the M-20 binder resin since similar chemistries are involved and the ratio of infusion resin to binder resin is relatively high. Infusion resin catalysis of the binder resin appears likely since the infusion resins can dissolve the small M-20 resin islands and significant static mixing occurs as the infusion resin percolates through the preform. The combination of thermal cure of M-20 and infusion resin catalysis of the binder suggests that the binder solutions can be formulated without the addition of the dinonyl phenol and the CoAcAc catalysts.

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A typical formulation for the 5250-4-RTM BMI binder solution is:

5250-4-RTM BMI Resin	80 % by weight
MEK	20 % by weight

To formulate this binder solution, the 5250-4-RTM BMI resin is heated and dispensed typically from a 5 gallon can using a Graco hot melt dispenser. The resin is heated to between about 230°F and 270°F prior to dispensing. At these temperatures, the BMI will slowly advance over a period of approximately 3 hours. Since the dispensing operation can be completed in less than 15 minutes, insignificant resin advancement occurs.

Insignificant advancement also occurs during the thermal vacuum dry out cycle. Once the resin is dispensed into a vacuum mix pot and has cooled slightly, MEK solvent is added to the resin. The resin is dissolved into the solvent carrier using a pneumatically driven stirrer in a sealed container. Pneumatic stirring is used to avoid potentially explosive conditions that could result with electric mixers. Once the resin is dissolved, the BMI binder solution is ready for use.

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Generally speaking, more care must be exercised when handling dry or binderized preforms relative to traditional prepreg materials. On the other hand, dry or binderized

preforms typically require fewer vacuum debulking [steps] steps than prepregs. For simple geometries, the entire stack of collated preform material layers can be vacuum debulked once at room temperature. As the part complexity, contouring, features, and thickness tailoring increase, additional vacuum debulking steps are required during the preform collation.

Binderized materials [ARC] are vacuum debulked at room temperature to produce “soft binder preforms.” Typically, soft binder preforms must remain on the forming tool for shape retention, but sometimes can be precision trimmed for net molding operations. The consolidated “soft binder preforms” tend to have some springback and tend to expand in thickness slightly when removed from a vacuum bag. The amount of “springback” after consolidation is dependent on the binder tack, the binder content, and other factors.

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Flow media 60 is then laid over the peel ply 59 (Fig. 2). The flow media should be a low profile material that has high, uniform permeability relative to the preform, such as open weave fiberglass, screening material, woven metallic screens, or chopped glass mats. The media should drape for contouring, have no contamination potential to the infusion resin, provide adequate stiffness to prevent bag mark offs on the part, and survive the required cure cycle. TEFLON-impregnated, open weave fiberglass materials such as Taconics 7195 or ChemFab CHEMGLAS 1589 perform particularly well as a flow media material. TEFLON-impregnated fiberglass materials are approximately 0.020 inch thick, have a uniform woven structure, are chemically inert, and are resistant to temperatures up to 600°F. Their somewhat boardy (stiff) nature allows contouring and bending, but also serves to prevent bag mark off. Their permeability helps to control the infusion resin wave front and prevents trapped void formation during the infusion, but can create problems for infusions with resins having very high viscosities or limited working times before thickening. To increase the permeability of the media while still retaining the uniform feed and reduced mark off characteristics, higher permeability materials can be placed over the TEFLON-impregnated fiberglass. One option is to use coarser Taconics 8308 or simply another layer of Taconics 7195 over the Taconics 7195 to create a more permeable flow media combination which will dramatically speed infusion rates and allow processing of more viscous resin systems.

The inner bag 62 generally is a disposable or consumable film or elastomer. For long production runs, however, especially of complex structures, conformable, premolded, reusable elastomeric bags made from silicones, fluorosilicones, Fluorel, nitrile rubber or other elastomeric materials may be preferred. The bag 62 should be flexible and have high elongation capability with relatively low modulus to simplify bagging complex parts so that it can be vacuum formed around the preform even where bridged. Bag bridging can occur over the preform at discontinuities. A low modulus bag reduces localized bag stresses on the preform which otherwise can cause tapering, distortion, or preform damage. Shaping the bag to conform with the contour of the preform minimizes resin rich zones in the finished parts, resin channeling, and edge tapering from bag induced stresses. Although stiff at room temperature, the film may become sufficiently flexible to stretch when heated for vacuum dry out, infusion, or curing. Standard nylon bags for prepreg material processing at 350°F will work, but are not optimal because of their relatively low ultimate elongation (200-300%) and high stiffness. STRETCHLON 700 polyester and STRETCHLON 800 nylon bagging films from Airtech International are superior, because they can stretch over 500 % and [are more flexible] have lower modulus than the standard nylon films. A VACPAC polyurethane film from Richmond Products is effective for low temperature cures below 160°F, because it has an extremely low modulus at room temperature and an ultimate elongation approaching 1000 %. For cures up to 600°F, special FEP based bagging films, such as ChemFab's VB3, can be used instead of polyimide based films such as KAPTON or THERMALIMIDE. This FEP bagging material, [when] which is etched on one side to improve seal adhesion, has elongation over 500 %.

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Rubber molded bags are significantly more expensive than disposable bagging films, [but] and the bags tend to degrade faster than one would expect. Release liners are often attached to the molded rubber, but they can disbond and create spider wrinkles on the bag molding surface. Seam failures or bag tears can also occur.

Making the master mold for a rubber bag can be difficult and expensive. It must be sized to accommodate the high bag shrinkage that will occur after cure. [Nevertheless] Even then, the bags continue to shrink over repeated-cure-cycles resulting in poorer and

poorer fits with the preform. The assembly problem is compounded because the rubber bags have a much higher stiffness and loading on the relatively unstable preform than disposable film bags. Force fitting the bags can actually result in preform movement or damage. Properly cared for, the bags can only withstand about 100 cure cycles at 350°F, but they often fail in less than 10 cycles.

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A vacuum source 11 is connected to the drop out tank 9 to pull vacuum on the installed inner bag 62. The vacuum line typically has quick connect fittings on both ends allowing it to be easily attached to the drop out tank and vacuum source. Once the bag is pulled tight with vacuum, the vacuum level is checked with a precision, vacuum test gauge or vacuum transducer 12. If the part has an obviously low vacuum level as indicated by the gauge or signals from the vacuum pump, the bag and connections are checked with a leak detector until the leak is found and repaired. With a high efficiency vacuum pump, the vacuum level should consistently exceed 28 inches of Hg. A vacuum in excess of 29 inches of Hg is preferred because it provides additional preform compaction.

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o. Are [Low] low cost with high temperature performance for demanding applications;

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Preferred resins include Bryte Technologies EX-1510 and EX-1545, cyanate esters, ATARD Laboratories SI-ZG 5A anhydride based epoxy, and Cytec-Fiberite's 823 epoxy. Preferred resins are low viscosity liquids at room temperature and consequently do not require heating for infusion. Some resins such as Ciba Geigy's 8611 are thick viscous liquids at room temperature (between say 1,000-10,000 centipoise) and must be heated to relatively low temperatures (between 100-160°F typically) to reach an acceptable viscosity (less than 400 centipoise). Other resins such as 3M's PR 500 and Cytec Fiberite's 5250-4-RTM are semi-solids at room temperature and must be melted at relatively high temperatures to infuse in an acceptable viscosity range. We shy away

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Page 44, 3d paragraph

Semi-solid materials are most efficiently dispensed and heated using a Graco hot melt dispenser. These resins are normally de-aired in heated vacuum ovens to minimize cooling.

Page 44, last paragraph

The weight of the preform can be measured directly or be estimated from known preform ply areas and nominal areal weights. With the resin and preform weights and [density] densities (i.e., specific gravity), resin content and fiber volume can be easily determined.

Page 45, 2d & 3d

When the resin must be infused at elevated temperature onto a hot tool, the operation must be performed in an oven or the tool and /or resin must be heated. For ambient processing, the infusion can be performed in practically any convenient location or in the oven directly. If the infusion is performed outside the oven, processing capacity is maximized. When infusions are performed outside the oven, it is important that the vacuum level in the outer bag does not decrease during the transfer from the infusion site to the oven for cure.

To begin the infusion, the end of the feed line 8 is cut with a tube cutter to remove the portion of the tube with the sealant plug. ~~An external constricting device is installed~~

on the feed tube to reduce the flow rate of the resin in the initial phase of the infusion. Without this feed constraint, the resin tends to shoot into the part too rapidly and can trap voids behind the wave front that are difficult to remove. The end of the feed tube is placed in the feed container 14 near the base and is secured. The feed can may be tilted at an angle with the feed tube positioned in the lowest location to minimize the amount of resin required to prevent air from entering the tube and bagged preform. To initiate flow, the sheet metal or welding clamp is removed from the feed tubing. After a few minutes of infusion, the constricting device is normally removed from the feed tube to speed the infusion rate.

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The feed [can] should be positioned below the lowest part of the preform. Positive pressure feed to the preform causes the inner bag to bulge near the feed spring. The vacuum tubes, on the other hand, should rise above the preform to help maintain hydrostatic pressure on the fluid and to minimize resin drain from the preform into the drop out can. Although preforms can be infused successfully in the horizontal orientation, it is often preferable to infuse in an inclined or vertical orientation with the feed at the lowest end and the vacuum pulled at the highest end. Inclined or vertical orientations tend to reduce channeling effects in low viscosity resin systems and in preforms with high variations in permeability. These orientations can also be used to eliminate otherwise necessary plumbing.

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The vacuum lines should be throttled or choked to a near closed position until the mass flow rate of resin through the preform equals the mass flow rate in the vacuum tube. In the choked condition, the resin feed to the preform and the tubes exceed the pull off capability downstream of the choke point. Consequently the preform will completely fill. As the preform fills, the mass flow rates in the preform will eventually decrease to match the mass flow rate beyond the choke point. Once this quasi-steady state is reached where the feed and pulloff rates are equal and the preform is full, the bubbling action associated with the fill and drain phenomena ceases. The vacuum tube between the preform and the tube choke point eventually fills with bubble free resin. The system normally reaches a quasi-steady state after approximately 15 minutes of choke flow processing.

Another approach that can be used to prevent preform draining is to regulate the vacuum on the inner bag. Reducing the vacuum level reduce[d]s the flow rates in the tubes. The preform has a lower tendency to drain, especially for more viscous resins that have sufficient body to move through a preform as a continuous pool. The resin also has little tendency to separate into discrete fluid bodies. Using this approach, the inner bag vacuum level is typically dropped from 29+ inches Hg to 22 - 27 inches Hg. Shortly after dropping the vacuum level, the bubbling will stop as with the throttling devices. A problem with this approach is that the inner bag may move[s] toward the outer bag because of the reduced vacuum. Movement decreases preform compaction and ultimately produces lower fiber volume composites.

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After the preform is completely filled with resin and the resin flow rate is constant, the feed and vacuum tubes are clamped closed simultaneously with sheet metal or welding pliers. The vacuum source is disconnected from the drop out tank. Both vacuum tubes and the feed tube are cut near the welding pliers. The resin in the vacuum tubes is sucked into the drop out tank and the resin in the feed tube drains into the feed container. The process results in complete resin reclamation and allows [near] real time mass balances to be performed. The ends of the cut vacuum tubes and feed tubes are sealed with pressure-sensitive adhesive tape and then wrapped with vacuum bag sealant tape. The tube seals are simply a redundant measure to prevent air from entering the inner bag in the event the welding pliers fail to isolate the inner bag from atmospheric pressure. Prior to beginning cure, the bulk resin in the feed can and the drop out can is removed from the oven to prevent unwanted hazardous exothermic reaction. Likewise, all other tool and consumable materials are removed prior to closing the oven for cure.

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Our preferred process allows resin recycling or recirculation. In some complex infusions where, for example, separate wave fronts converge together, extra resin may need to be purged from the preform to remove[d] trapped air or voids. Resin recirculation rather than continuous purging minimizes resin waste and expense. With recirculation, excess resin is typically charged to the system to have a reasonable working volume. The resin is allowed to accumulate in the drop-out can. Once the resin in the

supply can begins to run low, the feed and vacuum tubes are clamped shut. The vacuum source to the drop out tank is disconnected and the vacuum is released using the quick connect fittings. With the vacuum released, the lid on the drop out tank can be removed, and resin drained into the can from the tubes. The resin in the drop out can is transferred to the source container. Sufficient time, usually about 5 minutes, is given to allow entrained air to percolate out of the resin before flow is reinitiated. The drop out tank is reassembled and evacuated with a separate isolated vacuum pump to prevent any possible vacuum decrease in the outer bag. Once the tank is at the original vacuum level, all tube clamps are released simultaneously establishing flow again. The process can be repeated until all voids and bubbles are removed from the preform. At this point, the infusion can be terminated.

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Once the infusion is completed, high vacuum must be maintained in the outer bag of the infused preform throughout the cure cycle, especially immediately before and during resin gelation. Vacuum loss during this critical stage will cause the inner bag to relax, [that will increase] increasing the volume in the inner bag. The infused preform will swell because resin cannot be added in the closed system, the swelling reduces the hydrostatic pressure which produces surface porosity and voids, reduced preform compaction, and lower fiber volumes.

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The composites can, then, be inspected using any one or all of many non-destructive inspection (NDI) techniques of the type typically used to inspect autoclave-cured composites, including ultrasonic and radiographic techniques. Inspection may be avoided if certain in-process controls are used throughout the manufacturing process. Visual observation of the flow through windowing in the breather material, use of optically clear tooling made, for example, from PYREX or LEXAN, and optically clear or translucent tubing provide indications of quality during the infusion. Similarly, mass balances, infrared flow front detection, imbedded, remotely queried sensors, or flush tool mount sensors can provide in-process indications of quality. Visual inspection of the laminates after processing generally is a good indicator of their quality. If the laminates do not have surface porosity (particularly on the tool side), if the thickness is within nominal limits, and if the composite rings when "coin tapped" (see, e.g., US Patent Application 08/944,885), the laminates will likely pass ultrasonic inspection. If any surface voids appear on

the parts, ultrasonic inspection is warranted. Because we have determined that there is a strong correlation between the existence of surface voids and the overall composite quality, simple inspection for surface voids can significantly reduce or even eliminate more sophisticated inspection using expensive ultrasonic, laser, or radiographic processes.

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The outer bag in the double bag system reduces thermal oxidation of the inner bag. This translates into a stronger bag that is less susceptible to leakage during cure at high temperatures. The outer bag and breather buffer the inner bag from handling damage that can occur in many ways. The outer bag applies pressure to the inner bag seals and improves the sealing effectiveness of those seals. The pressure on the inner bag seals overcomes bag peel stresses that can open up leaks, particularly at pleated seal locations. Because the outer bag encapsulates the inner bag, seals can not be worked loose in the convective environment typically found in ovens.

If a leak should occur in the inner bag, the result is not necessarily catastrophic as it generally is for single bag infusions. A leak in the inner bag will cause resin to flow into the outer bag. Corrective actions are possible with accelerated cures and bleed control techniques. A ruptured bag in a single bag environment allows air to enter the bag. The bag can swell and porosity can be continuously introduced into the laminate, resulting in catastrophic failure. Bag integrity differences between single bag infusion techniques and double bag techniques may not be significant when producing small, simple, low value composites. When attempting to produce large and/or complex composite assemblies, such as composite wings, the significance of the integrity differences is dramatically amplified. It is wise and prudent to use the double bag technique over single bags when producing these types of structures. Yield, integrity, and process robustness become far more important factors in reducing overall cost than eliminating the cost associated with a second bag.